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THE ISOTOPIC CONSTITUTION OF TUNGSTEN, SILICON AND BORON

A DISSERTATION SUBMITTED TO THE
FACULTY OF THE DIVISION OF THE
PHYSICAL SCIENCES IN CANDIDACY FOR
THE DEGREE OF DOCTOR OF PHILOSOPHY

DEPARTMENT OF PHYSICS

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Reprinted from THE PHYSICAL REVIEW, Vol. 70, Nos. 9 and 10, November 1-15, 1946

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The Isotopic Constitution of Tungsten, Silicon, and Boron

MARK G. INGRAM

Department of Physics, University of Chicago, Chicago, Illinois

(October 1, 1945)

The isotopic constitutions of tungsten, silicon, and boron have been determined. Five isotopes of tungsten were observed at masses 180, 182, 183, 184, and 186 with abundances of 0.122 percent, 25.77 percent, 14.24 percent, 30.68 percent, and 29.17 percent, respectively. Three isotopes of silicon were observed at masses 28, 29, and 30 with abundances of 92.28 percent, 4.67 percent, and 3.05 percent, respectively. Two isotopes of boron were observed at masses 10 and 11 with abundances of 18.83 percent and 81.17 percent, respectively. Upper limits for the existence of other hypothetical normal isotopes in the immediate neighborhood of the known isotopes are given.

INTRODUCTION

THIS paper is a report on a mass spectrometric study of the isotopes of tungsten, silicon, and boron. This study was undertaken to: verify the existence of the rare mass 180 isotope of tungsten, to make accurate determinations of the isotopic constitution of these elements, and to search the mass numbers in the immediate vicinity of the known isotopes for possible rare isotopes and, if they cannot be detected, to place upper limits for the existence of these hypothetical isotopes.

APPARATUS

The mass spectrometer used in this investigation was a Nier type, 60°, 6-inch radius of curvature, single focusing mass spectrometer.¹ The gas to be analyzed was introduced through a capillary leak to the ionization chamber of the source, and ionized by a 0.5-ma beam of electrons of 85-volts energy. The ions formed were collimated into an ion beam of 2000-volt energy by the collimating plates of the source, and directed normally into a wedge-shaped magnetic field. This magnetic field resolved the ion beam into its various mass components and refocused a beam of one mass component through the collector slit to a collector plate. The collector plate was surrounded by an electron repelling field of 22½ volts, so that secondary electrons formed at the collector plate by positive ion bombardment could not leave that plate. Ion currents were measured by either of two methods: (1) the conventional FP-54 electrometer tube method² in

which the voltage developed across the input resistor is canceled by an equal and opposite voltage supplied from a Leeds and Northrup type K-2 potentiometer, or (2) the 100 percent inverse feedback method³ in which the amplifier drives a standard Brown Electronik Strip Chart Recorder so that permanent records of actual peak shapes are obtained. The latter of these methods was used for the major portion of the work here reported, and the first only as a check.

The pumping system consisted of a two-stage, glass, mercury diffusion pump, backed by a Welch Duo-Seal fore pump. A liquid air trap was used between the diffusion pump and the mass spectrometer tube to trap out vapors. The pumping speed of the system at the tube was 2.5 liters per second. The mass spectrometer tube was wrapped with a furnace so that the entire system could be baked out at a temperature of 450°C. With proper precautions this system could be maintained at 1×10^{-7} mm of mercury pressure.

DISCUSSION OF ABSOLUTE MEASUREMENTS

Before proceeding to the results, some of the possible systematic errors which bear on the interpretation of absolute isotopic measurements will be discussed.

Magnetic Field Variation

Either of two basic methods can be used to bring the ion beam of desired mass to focus on the fixed collector. This can be seen by referring

¹A. O. C. Nier, *Rev. Sci. Inst.* 11, 212 (1940).

²D. B. Penick, *Rev. Sci. Inst.* 6, 115 (1935).

³A. O. C. Nier, E. P. Ney, and M. G. Ingram, to be published.

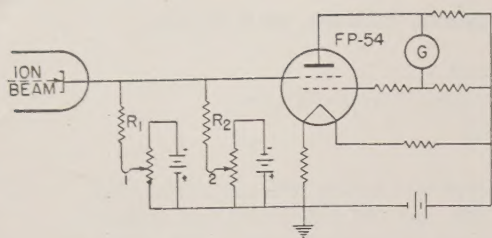


FIG. 1. Circuit used for determining the degree of non-linearity of high resistors in the low voltage range.

to the familiar Eq. (1) which gives the radius of curvature of the ions in a magnetic field.

$$\frac{m}{e} = 4.82 \times 10^{-5} \frac{H^2 r^2}{V} \quad (1)$$

In this equation m is the mass of the ion in atomic mass units, e is the number of electronic charges on the ion, H is the magnetic field in gauss, r is the radius of curvature of the ions in the magnetic field in cm, and V is the energy of the ions in volts. Thus to bring ions of different m/e to focus on the fixed collector, i.e., fixed r , either H or V must be changed. With the machine used in the present investigation, measurement of the isotopic ratio by changing the ion energy gave, for conditions of poor focus in the source, variations as large as ± 20 percent for a 10 percent difference in mass. This variation is explained as caused by a change in efficiency of the source, as the voltage across it is changed. On the other hand, measurements of the isotopic ratio when the ion trajectories were changed by varying the magnetic field varied by less than ± 0.5 percent for a 10 percent difference in mass for widely different fields. For this reason, all measurements reported in the present work were obtained by varying the magnetic field to bring the different mass ions to focus on the fixed collector.

To verify that stray magnetic fields in the source region had no influence on the results, isotopic ratios were obtained with and without a 300-gauss magnetic field around the source. The maximum difference observed was one percent in the ratio for a 10 percent difference in mass. Since the stray field from the main magnet was always less than $\frac{1}{10}$ of this test 300-gauss field, it may be concluded that when no source magnet is used the systematic error introduced by stray magnetic fields in the source region should not exceed 0.1

percent of the ratio for a 10 percent difference in mass. In all cases where sufficient ion intensity could be obtained without the use of a source magnet, the magnet was not used.

Ion Measurements

To check possible non-linearities in the ion measurement circuits two methods of ion measurement were used. In using the FP-54 electrometer² the voltage developed across the input resistor was cancelled by an equal and opposite voltage supplied by a type K-2 potentiometer, so that the ratio of any two ion beams was determined as the ratio of the voltages supplied by the potentiometer to cancel out those ion currents. With the 100 percent inverse feedback amplifier and the Brown Electronik Strip Chart Recorder, the voltage developed across the input resistor was recorded by the recorder, and the ratio of any two ion currents was determined as the ratio of the two peak heights recorded on the recorder. Within the limits of experimental error, ± 0.3 percent, no systematic effect could be observed between the two methods of measurement. The major question remaining in the linearity of the detecting circuits is then the non-ohmic response of the input resistor which is common to both methods of measurement.

Linearity of Resistors

To measure the non-linearity of the high resistors (4×10^{10} ohms) in the low voltage range (0.001 to 10 volts) the circuit shown in Fig. 1 was devised. The operation of this circuit can be understood from the following considerations. If the potentials applied to the points 1 and 2 are zero with respect to ground, and a small ion current sufficient to produce one millivolt across the input resistors is allowed to strike the collector, a deflection D_1 will be observed on the galvanometer. If now a potential of one volt is applied to point 1, and minus one volt to point 2, the potential on the grid of the FP-54 will still be at ground. Now allowing the same ion current to fall on the collector as was used with the potentials of points 1 and 2 at ground, a second deflection D_2 of the galvanometer will be produced. The ratio of D_1 to D_2 will then be the ratio of the effective resistances of the high resistors at

0.001 and 1.00 volts, respectively, across the resistors. By repeating this procedure for other voltages applied to the points 1 and 2, a curve can be drawn for the effective resistance of a set of resistors for any voltage across them. By proper use of three resistors, the effective resistance of a single resistor, with any predetermined voltage across it, can be found. Using this method it was found that most IRC MG-6 resistors are ohmic in the range 0.001 to 10 volts within ± 0.2 percent of their value. Some other resistors exhibited marked non-linearities and polarizations in this range. The measurements reported here were made with resistors which showed no non-ohmic response.

Secondary Electrons

That the errors due to secondary electron emission from the collector plate were negligible can be seen from Fig. 2. This curve is a plot of the current measured by the collector as a function of the voltage supplied to suppress the secondary electrons. This curve shows that below -15 volt less than one percent of the total ion current was caused by secondary electrons. Since $22\frac{1}{2}$ volts was used as the suppressor voltage and the potential on the collector plate never changed by more than 0.01 volt in the present measurements, it may be concluded that the secondary electron contribution to the measured ion currents was less than one percent for all measurements here reported. Curves for the variation of secondary electron emission with the mass of the bombarding ion show that very roughly the emission varies by 0.7 times the variation in mass for the surfaces used in these experiments. We may conclude that the systematic error in isotopic ratio due to secondary electron emission should be less than 0.03 percent for a 10 percent difference in mass. This is completely negligible. The importance of suppressing secondary electrons can be seen from the fact that if they had not been suppressed, the error would have been 3 percent in the ratio for a 10 percent difference in mass.

Various Other Possible Errors

Systematic discriminations were looked for that might be caused by the various accelerating and focusing plates in the source region. The maximum variation obtained, with wide changes

in the potentials of these plates, when no source magnet was used, was 0.3 percent of the ratio for a 10 percent difference in mass.

In view of possible space charge effects, the ratios were studied as a function of ionizing electron current. The variations observed when the currents were changed by a factor of 5 was less than 0.3 percent of the ratio for a 10 percent difference in mass.

A change of isotopic abundance due to passage of the gas through the leak is another factor which must be considered as a source of systematic error. In the cases of tungsten hexafluoride, silicon tetrafluoride, and boron trifluoride, the maximum change that would be expected in the isotopic ratio would be 1.00 percent, 0.95 percent, and 0.74 percent, respectively. In the operating region, the peak height in the spectrometer varied approximately as the pressure of the gas behind the leak to the 1.4 power. The fractionation of the leak should therefore be appreciably below the maximum.

In the case of the analysis of tungsten by using the gas tungsten hexafluoride, a small systematic error will be introduced if the concentration of the isotopes in the vapor phase is different from the concentration of the isotopes of the solid phase with which it is in equilibrium. The order of magnitude of this error is below a few tenths of one percent. The silicon tetrafluoride and the boron trifluoride were completely in the vapor phase so that no error could be introduced from this effect in these cases. The errors resulting

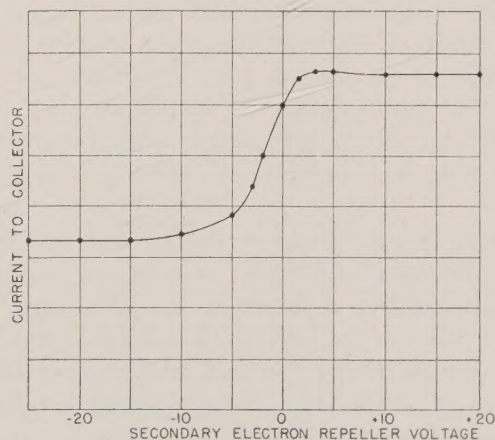


FIG. 2. Graph showing the energy and number of secondary electrons produced by collision of ions of 2000 volts energy with the collector plate.

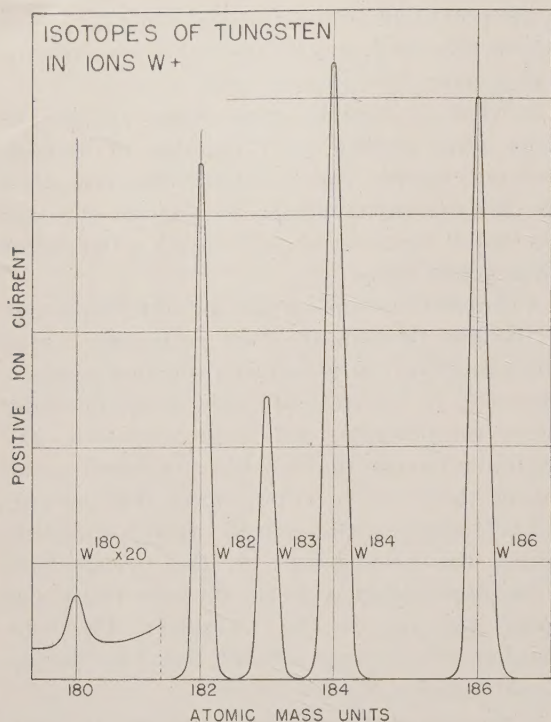


FIG. 3. Recorder tracing of the isotopes of tungsten as observed in the W^+ ion position. The region about mass 180 is recorded at 20 times the sensitivity used for the rest of the tracing.

from the chemical preparation of the samples cannot be evaluated, but are undoubtedly less than a few tenths percent.

If the peak shape for peaks of widely differing intensities are not identical, the slit systems could discriminate systematically according to the intensity of the peaks. To check the magnitude of this effect the Si^{28}/Si^{30} ratio was studied as a function of the collecting slit width. The errors observed were random, so that it may be concluded that within 0.2 percent of the ratio there is no error introduced due to differences in peak shape.

From consideration of the systematic error uncertainties it is felt that none of the absolute abundances here reported can be relied upon to better than $\pm$ one percent of the abundance of that isotope. Hence the final answers quoted in Tables IV, VIII, and XI, are quoted with the largest of the two errors, i.e., the standard deviation of the actual results or the systematic error uncertainty.

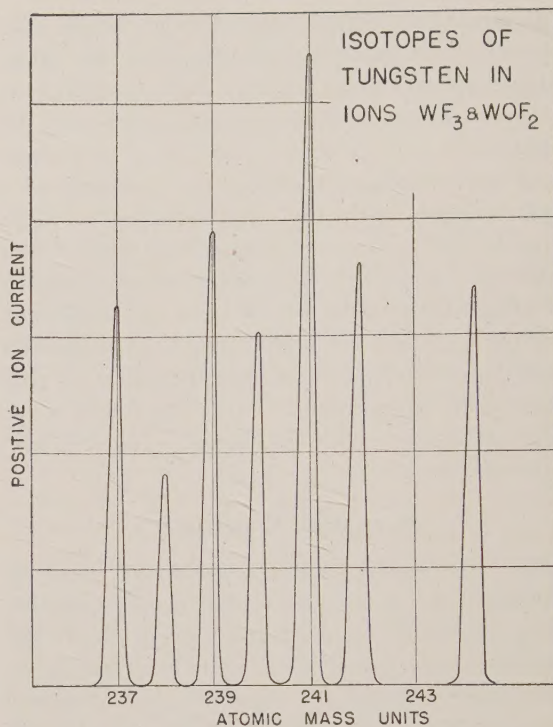


FIG. 4. Recorder tracing of the isotopes of tungsten as observed in the WF_3^+ position. The presence of the compound WOF_2^+ is apparent.

EXPERIMENTAL RESULTS

Tungsten

Tungsten was first studied by Aston⁴ using tungsten hexacarbonyl as the source of tungsten. By photographic methods he detected isotopes of mass 182, 183, 184, and 186 with abundances of 22.6 percent, 17.3 percent, 30.1 percent, and 29.8 percent, respectively. Later Dempster⁵ working with a spark source discovered a rare isotope of mass 180 and estimated its relative abundance as one percent of the 183 isotope, or approximately 0.17 percent.

In the present investigation the isotopic constitution was studied by using a mixture of WF_6 and WOF_4 . Figure 3 shows a curve obtained on the Brown recorder of the tungsten spectrum in the W^+ position, and Fig. 4 shows the same type of curve taken in the WF_3^+ position. The existence of the peaks due to WOF_2^+ is apparent in the latter. Calculations of the isotopic constitution can be made directly from the heights

⁴F. W. Aston, Proc. Roy. Soc. **A132**, 491 (1931).

⁵A. J. Dempster, Phys. Rev. **52**, 1074 (1937).

of the peaks in Fig. 3, and from Fig. 4 by resolving the spectra due to the two compounds. Using these techniques the isotopic constitution of tungsten was determined in the WF_5^+ , WF_4^+ , WF_3^+ , WF_2^+ , W^+ , WF_2^{++} , and W^{++} positions. The abundances obtained, for the moment neglecting the rare isotope at mass 180, are given in Table I. No accurate determinations were possible in the WF^+ or WF^{++} positions, due to mercury backgrounds which interfere in these positions. Similarly results in the WF_4^{++} , and WF_3^{++} positions were unreliable due to other large interfering impurities.

The data were taken over a three-week period. The consistency of any one run was much better than the comparison between runs. For this reason a direct average, rather than a weighted average, was used in determining the standard deviations given.

The rare isotope of tungsten at mass 180 discovered by Dempster⁵ was verified by studying the WF_5^+ , W^+ , WF_4^{++} , and W^{++} positions. It could not be determined accurately in the other positions due to interference of the WOF_n peaks. The results obtained are tabulated in Table II.

The existence of the rare isotope of tungsten at mass 180 is proved by the facts that, in all of the nine positions studied, a peak was present to about 0.122 percent which could not be accounted for by known impurities. It would be very unlikely that this would have been the case if the peak corresponding to the 180 mass observed had been an impurity. The fact that the mass is lighter than any other mass in the tungsten spectrum shows that it cannot be an impurity in which tungsten is a part. Another proof results from the observation that varying the electron accelerating potentials gave the same appearance potentials for the 180 mass as for the other four

TABLE I. Abundances of tungsten isotopes other than rare isotope at 180.

Ion studied	% 182	% 183	% 184	% 186	No. of measurements
WF_5^+	26.02	14.22	30.74	29.00	68
WF_4^+	25.80	14.14	30.89	29.17	10
WF_3^+	25.73	14.46	30.54	29.24	12
WF_2^+	25.83	14.34	30.88	29.00	20
W^+	25.76	14.14	30.83	29.26	97
WF_2^{++}	25.57	14.41	30.59	29.41	57
W^{++}	25.87	14.09	30.71	29.44	31
Average	25.80 ± 0.13	14.26 ± 0.14	30.74 ± 0.13	29.22 ± 0.16	

isotopes of tungsten. It must therefore be concluded that tungsten has a stable isotope of mass 180.

The regions in the immediate neighborhood of the known isotopes were searched for other possible rarer isotopes. In some cases peaks were found which could correspond to unknown rare isotopes, but in every case a search in the other ion positions showed that these peaks were impurities. Table III gives the upper limits set on the existence of hypothetical isotopes together with the ion position in which this limit was determined. The fourth column of this table gives the tentative mass assignments given the artificially produced radioactive isotopes of tungsten as given by Seaborg.⁶

Thus, the isotopic constitution of tungsten as determined in the present work as compared with those given by Aston and Dempster is given in Table IV. The errors given are the systematic error uncertainties.

Calculating the chemical atomic weight from these abundances using the value of 1.8×10^{-4} for

TABLE II. Abundance of W^{180} .

Ion studied	% 180	No. of measurements
WF_5^+	0.119	16
W^+	0.121	16
WF_4^{++}	0.124	16
W^{++}	0.122	25
Average	0.122 ± 0.002	

TABLE III. Upper limit for abundances of tungsten isotopes not found.

Mass No.	Upper limit on existence	Ion studied	Artificial radioactivity ^a
174	0.001%	WF_4^+	
175	0.001	WF_4^+	
176	0.001	WF_4^+	
177	0.001	WF_4^+	
178	0.002	WF_4^+	
179	0.005	W^+	
181	0.01	W^{++}	
		W^{++}	
185	0.01	W^{++}	β^- , 77 day
187	0.02	WF_5^+	β^- , 24 hour
188	0.006	WF_6^+	
189	0.002	WF_6^+	
190	0.002	WF_6^+	
191	0.001	WF_6^+	
192	0.001	WF_6^+	

^a See reference 6 in text.

⁶ G. T. Seaborg, Rev. Mod. Phys. 16, 1 (1944).

the packing fraction of tungsten⁷ and the factor 1.000275 in converting from the physical to the chemical atomic weight gives 183.89, which is in good agreement with the chemically determined value of 183.92.

The relative intensities of the various ion fragments observed in this mass spectrometer when WF_6 was bombarded by electrons of 85-volts energy are given in Table V.

Silicon

Silicon was first studied by Aston⁸ using the ions formed in SiF_4 gas. He showed with the photographic method that the main isotope had a mass of 28 and that there was a second weak isotope at mass 29. He noted a line at mass 30, but the possibilities of hydrogen compounds made this inconclusive. Later⁹ he proved the existence of the mass 30 isotope during his work on zirconium. The latest determination of the isotopic constitution is that of McKellar¹⁰ in which he used photometry on the bands of SiN . He gives the abundances of masses 28, 29, and 30 as 89.6 percent, 6.2 percent, and 4.2 percent respectively. No limits have been set on the existence of other hypothetical normal isotopes of silicon.

The isotopic constitution of silicon was determined in this investigation by studying the

TABLE IV. Isotopic constitution of tungsten.

Investigator	%180	%182	%183	%184	%186
Aston	—	22.6	17.3	30.1	29.8
Dempster	~0.17	—	—	—	—
Present work	0.122 ±0.002	25.77 ±0.3	14.24 ±0.2	30.68 ±0.3	29.17 ±0.3

TABLE V. Relative intensities of ion fragments when WF_6 is bombarded by 85 ev electrons.

Ion	Intensity	Ion	Intensity
WF_6^+	0.5	WF_6^{++}	0.5
WF_5^+	100	WF_5^{++}	0.5
WF_4^+	22	WF_4^{++}	20
WF_3^+	32	WF_3^{++}	17
WF_2^+	51	WF_2^{++}	16
WF^+	36	WF^{++}	15
W^+	33	W^{++}	14

⁷ A. J. Dempster, Phys. Rev. **53**, 869 (1938).

⁸ F. W. Aston, Phil. Mag. **40**, 628 (1920).

⁹ F. W. Aston, Phil. Mag. **49**, 1198 (1925).

¹⁰ A. McKellar, Phys. Rev. **45**, 761 (1934).

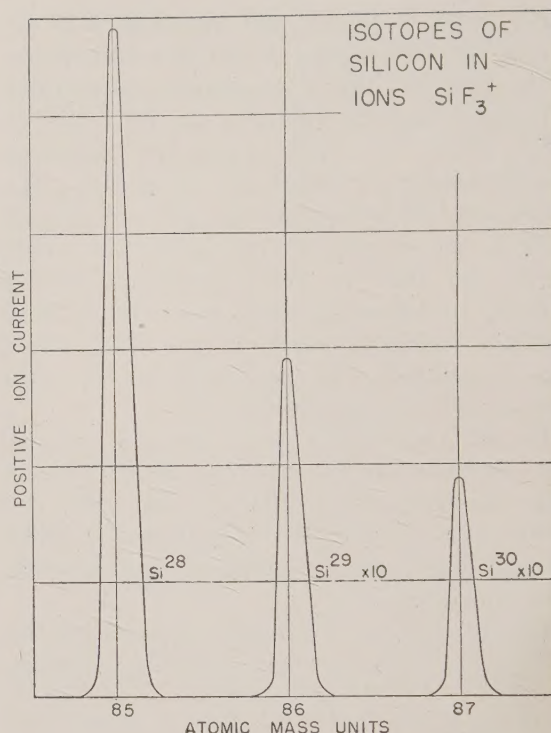


FIG. 5. Recorder tracing of the isotopes of silicon as observed in the SiF_3^+ position. The region about masses 86 and 87 are recorded at ten times the sensitivity used in recording the 85.

SiF_3^+ ions formed by electron bombardment of SiF_4 gas. The other positions are unreliable due to impurities of boron trifluoride, carbon dioxide, and nitrogen. Figure 5 is a recording taken on the Brown recorder of the silicon isotopes in the SiF_3^+ position. Table VI gives the isotopic composition of silicon as determined in this position by the two different measurement techniques. The errors given with the averages are the standard deviations.

Following the method described for establishing lower limits on the existence of rare isotopes of tungsten, the results obtained for silicon are given in Table VII.

The isotopic constitution determined in the

TABLE VI. Isotopic composition of Si.

Method	28	29	30	No. of measurements
Recording spectrometer	92.31	4.67	3.02	17
FP-54 Electrometer	92.25	4.68	3.08	10
Average	92.28 ± 0.03	4.67 ± 0.01	3.05 ± 0.03	

present work compared with the values of McKellar are given in Table VIII. The errors given are the systematic error uncertainties.

It should be noted that the ratio of the 29 to the 30 obtained in the present work is very close to that obtained by McKellar; the main difference in the two determinations is in the ratio of the two small isotopes to the 28.

Calculating the chemical atomic weight from the abundances using the packing fractions of 4.86×10^{-4} , 4.54×10^{-4} , and 5.68×10^{-4} for the masses 28, 29, and 30, respectively, as given by Pollard¹¹ and the factor 1.000275 in converting from the physical to the chemical atomic weight gives 28.086 which is in good agreement with the chemically determined value of 28.06.

Boron

Boron was first studied by Aston using the ions characteristic of BF_3 gas.¹² He noted two masses at mass 10 and 11. Since that time there have been several other investigations of the isotopic ratios using photometry methods.¹³⁻¹⁵ The latest value was obtained by Ornstein and Vreeswijk.¹⁶

In the present investigation the isotopic constitution of boron was determined by using the ions formed by electron bombardment of BF_3 and

$\text{BO}(\text{CH}_3)_3$. The latter material is more convenient to handle in the mass spectrometer since it does not form addition compounds with the walls of the spectrometer as readily as does BF_3 . The source of boron used was tank BF_3 supplied by the Harshaw Chemical Company. The results of the analyses are tabulated in Table IX.

Following the method used for establishing upper limits on the existence of hypothetical isotopes of tungsten, the upper limits set on the existence of possible rare isotopes of boron are tabulated in Table X.

The intensities of the peaks observed in BF_3 when bombarded by electrons of 85 volts energy are 3.8, 5.6, 90.5, and 0.2 for the B^+ , BF^+ , BF_2^+ , and BF_3^+ , respectively.

Thus the isotopic composition as determined in this work as compared with the results of other investigators is given in Table XI. The errors

TABLE IX. Isotopic constitution of boron.

Ion studied	% 10	% 11	No. of measurements
BF_3^+	18.89	81.11	10
BF_2^+	18.83	81.17	20
BF^+	18.87	81.13	10
B^+ from BF_3	18.73	81.27	20
B^+ from $\text{BO}(\text{CH}_3)_3$	18.81	81.19	20
Average	18.83 ± 0.06	81.17 ± 0.06	

TABLE VII. Upper limits of abundances of Si isotopes not found.

Mass No.	Upper limit on existence	Ion studied	Artificial radioactivity ^a
24	0.002%	SiF_3^+	
25	0.003	SiF_3^+	
26	0.006	SiF_3^+	
27	0.006	SiF_3^+	β^+ , 4.9 sec.
31	0.005	SiF_3^+	β^- , 170 min.
32	0.003	SiF_3^+	
33	0.002	SiF_3^+	
34	0.002	SiF_3^+	
35	0.002	SiF_3^+	

TABLE X. Upper limits for abundances of isotopes of boron not found.

Mass No.	Upper limit on existence	Ion studied	Artificial radioactivity ^a
7	0.003%	B^+	
8	0.003	B^+	
9	0.006	B^+	
12	0.003	BF_2^+	β^- , 0.022 sec.
13	0.0003	BF_2^+	
14	0.0003	BF_2^+	

^a See reference 6 in text.

TABLE VIII. Isotopic constitution of silicon.

Investigator	%28	%29	%30
McKellar	89.6	6.2	4.2
Present work	92.28 ± 0.08	4.67 ± 0.05	3.05 ± 0.03

TABLE XI. Isotopic constitution of boron.

Investigator	Method	%10	%11
Aston ^a	Mass spectra	19.8	80.2
Elliot ^b	Band spectra	21.6	78.4
Paton and Almy ^c	Band spectra	17.1	82.9
Ornstein and Vreeswijk ^d	Line spectra	18.4	81.6
Present work	Mass spectra	18.83 ± 0.2	81.17 ± 0.2

^a Reference 13 in text.

^b Reference 14 in text.

^c Reference 15 in text.

^d Reference 16 in text.

¹¹ E. Pollard, Phys. Rev. **57**, 1186 (1940).

¹² F. W. Aston, Phil. Mag. **40**, 628 (1920).

¹³ F. W. Aston, Proc. Roy. Soc. **A132**, 490 (1931).

¹⁴ A. Elliot, Nature **126**, 845 (1930).

¹⁵ Paton and Almy, Phys. Rev. **37**, 1710 (1931).

¹⁶ Ornstein and Vreeswijk, Zeits. f. Physik **80**, 57 (1933).

quoted for the present work are the systematic error uncertainties.

Calculating the chemical atomic weight from the abundances determined in this investigation using the packing fractions determined by Bainbridge¹⁷ of 16.3×10^{-4} and 11.8×10^{-4} for the masses 10 and 11, respectively, and the factor

1.000275 in converting from the physical to the chemical atomic weight, gives 10.821 which is in good agreement with the chemically determined value of 10.82.

The author takes pleasure in acknowledging his indebtedness to Professor A. J. Dempster and Professor A. O. C. Nier for their suggestions, and to B. M. Rustad and J. L. Curtain for their assistance in this work.

¹⁷ K. T. Bainbridge, *Phys. Rev.* **51**, 385 (1937).

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